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(19) (CA) **CANADIAN PATENT** (12)

(54) GRAVITY SETTLING

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GRAVITY SETTLING

Abstract of the Disclosure

Solids are separated from a liquid in a gravity settler provided with inclined solid intercepting surfaces to intercept the solid settling path to coalesce the solids and increase the settling rate.

This invention relates to liquid-solid separation, and more particularly, to liquid-solid separation by gravity settling.

In many operations, there is a need for effecting separation of solids from a liquid. Thus, for example, in U.S. Patent No. 3,856,675 there is disclosed an improved process for separating insoluble material from a coal liquefaction product wherein such insoluble material is separated by gravity settling in the presence of a promoter liquid having specific characteristics. In such a process, and similar processes, there is a need for improved apparatus for effecting such gravity settling.

The present invention makes it possible to provide for improved separation of insoluble material from a coal liquefaction product by gravity settling.

In accordance with one aspect of the present invention, there is provided a process for separating solids from a liquid by gravity settling wherein a liquid-solid mixture is introduced into a gravity settler at multiple locations and the settling paths of the solids are intercepted in the gravity settler by causing the solids to come into contact with intercepting surfaces to coalesce the solids and increase the solids settling rate.

In accordance with another aspect of the present invention, there is provided gravity settler for effecting liquid-solid separation in which the gravity settler is provided with intercepting surfaces which are slanted at an angle with respect to horizontal to intercept the settling path of the solids. The intercepting surfaces divide the interior of the gravity settler into at least two gravity settling zones and a fresh feed inlet means introduces solids dispersed in a liquid into at least two of the gravity settling zones. Each gravity settling zone is provided with an overflow outlet for withdrawing an essentially solids free liquid, and an underflow outlet for withdrawing solid containing liquid.

The invention will be further described with respect to the following drawings; wherein:

Figure 1 is a simplified schematic drawing of an embodiment of the gravity settler of the present invention;

Figure 2 is a simplified drawing of another embodiment of the gravity settler of the present invention; and

Figure 2A is a detailed view of the pipe connection for the embodiment of Figure 2.

An embodiment of the present invention is illustrated in Figure 1 wherein a gravity settling vessel 101, having a cylindrical upper portion 102 and a conically shaped lower

portion 103, is divided into a plurality of separate liquid-solid separation zones or chambers by inclined solid intercepting surfaces in the form of generally A-shaped plates 104 having oppositely slanted portions 105 and 106, with each of the oppositely slanted portions being sloped at an angle with respect to horizontal of from 45° to 75°. The A-shaped plates 104 define therebetween two liquid-solid separation chambers 107 and 108. Each of the separate solid separation chambers 107 and 108 is provided with an underflow outlet 109 and 110, respectively, with a combined overflow being withdrawn from the upper portion of each of the chambers 107 and 108, with such overflow withdrawal means being schematically indicated as 111. The outlet withdrawal means 111 may be comprised of a plurality of riser pipes which discharge into each other. Each of the separate chambers 107 and 108 is provided with a fresh feed inlet for introducing a liquid-solid feed, with such feed inlets being schematically represented at 112 and 113. The plates 104 are preferrably corrugated in that the corrugations provide additional surface for coalescing of solids and provide additional strength to the plates.

In operation, a liquid-solid feed is introduced into the gravity settler 101 through fresh feed inlets 112 and 113, and as the solids settle, they strike the slanted plates, thereby intercepting their settling path forcing them to coalesce and separate faster. The agglomerated particles, as a result of the inclined surface of the plates 104, move to the underflow outlets 110 and 109 respectively for final withdrawal from the gravity settler 101. It is noted that the interior wall of the vessel in the lower conical portion 103 thereof also functions as slanted plates to intercept the settling path of solids in the lowermost settling zone.

The essentially solid free liquid is withdrawn from the upper portions of the separation zones for ultimate withdrawal from the gravity settler 101 through overflow outlet 111.

Although the embodiment has been particularly described with respect to separate underflow withdrawal from each of the chambers, it is to be understood that underflow withdrawal may be effected in a combined manner such as by using a plurality of nested downcomer pipes. Similarly, overflow withdrawal can be effected other than as particularly described. As a further modifiction, the intercepting surfaces may take the form of V-shaped plates whereby the intercepting surfaces are in the form of superimposed cones.

As hereinabove described, the gravity settler of the present invention is particularly suitable for separating insoluable material from a coal liquefaction product wherein the gravity settling is effected in the presence of a promoter liquid.

The coal liquefaction product is introduced into the gravity settler in admixture with a promoter liquid, as net feed, with the promoter liquid having the properties described in U.S. Patent No. 3,856,675. As described in the aforementioned patent, the promoter liquid is one that has an aromaticity less than that of the liquefaction solvent and is generally a hydrocarbon liquid having a characterization factor (K) of at least about 9.75 and preferably at least about 11.0, with such characterization factor being an index of the aromaticity/parafinicity of hydrocarbons and petroleum fractions as disclosed by Watson and Nelson, Ind. Eng. Chem. 25 880 (1933). The liquid

which is used to enhance and promote the separation of insoluble material is further characterized by a 5 volume percent distillation temperature of at least about 250°F and a 95 volume percent distillation temperature of at least about 350°F and no greater than about 750°F. The promoter liquid preferably has a 5 volume percent distillation temperature of at least about 310°F and most preferably of at least about 400°F. The 95 volume percent distillation temperature is preferably no greater than about 600°F. The most preferred promoter liquid has a 5 volume percent distillation temperature of at least about 425°F and a 95 volume percent distillation temperature of no greater than about 500°F.

As representative examples of such liquids, there may be mentioned: kerosene or kerosene fraction from paraffinic or mixed base crude oils; middle distillates, light gas oils and gas oil fractions from paraffinic or mixed based crude oils; alkylbenzenes with side chains containing 10 or more carbon atoms; paraffinic hydrocarbons containing more than 12 carbon atoms; white oils or white oil fraction derived from crude oils; alpha-olefins containing more than 12 carbon atoms; fully hydrogenated naphthalenes and substituted naphthalenes; propylene oligomers (pentamer and higher); tetrahydronaphthalene, heavy naphtha fractions, etc. The most preferred liquids are kerosene fractions; white oils; fully hydrogenated naphthalenes and substituted naphthalenes.

The amount of liquid promoter used for enhancing and promoting the separation of insoluble matter from the coal liquefaction product will vary with the particular liquid employed, the coal liquefaction solvent, the coal used

as starting material and the manner in which the liquefaction is effected. As should be apparent to those skilled in the art, the amount of liquid promoter used should be minimized in order to reduce the overall costs of the process. It has been found that by using the liquid of controlled aromaticity, the desired separation of insoluble material may be effected with modest amounts of liquid promoter. In general, the weight ratio of liquid promoter to coal solution may range from about 0.2:1 to about 3.0:1, preferably from about 0.3:1 to about 1.5:1. In using the preferred promoter liquid of the present invention which is a kerosene fraction having 5 percent and 95 percent volume distillation temperatures of 425°F and 500°F respectively, promoter liquid to coal solution weight ratios in the order of 0.4:1 to 0.6:1 have been particularly successful. It is to be understood, however, that greater amounts of liquid promoter may be employed, but the use of such greater amounts is uneconomical. In addition, the use of an excess of liquid promoter may result in the precipitation or separation of an excessive amount of desired coal derived products from the coal extract. More particularly, as the amount of liquid promoter employed is increased, a greater amount of ash is separated from the coal solution, but such an increased separation is accompanied by an increased separation of desired coal derived products from the coal solution. The net coal product (the extracted carbonaceous matter, excluding the promoter liquid, liquefaction solvent and gas make) contains less than about 1% insoluble material, generally less than 0.1% insoluble material, and most preferably less than 0.05% insoluble material, all by weight.

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The gravity settling is generally effected at temperatures from about 300°F to about 600°F, preferably from about 350°F to about 500°F, and a pressure from about 0 psig to about 500 psig, preferably at a pressure from about 0 psig to about 300 psig. It is to be understood, however, that higher pressures could be employed, but as should be apparent to those skilled in the art, lower pressures are preferred.

A modification of the embodiment of Figure 1 is illustrated in Figure 2 wherein the A-shaped plates are curved plates, formed from two upright conical sections 301 and 302 having the respective apexes 303 and 304 positioned at the vessel wall. The conical sections 301 and 302 are arranged such that they intersect with each other in the interior of the vessel above the cone apexes to form a curved or saddle-like intersection 305, with apexes 303 and 304 of the respective conical sections 301 and 302 being spaced 180° from each other. Thus, the internal plates are defined by the intersection of two vertical conical sections and the vessel (preferably a cylindrical vessel), with the apex of each cone being spaced on the vessel wall 180° from each other.

The vessel is provided with at least two vertically spaced intersecting conical sections, with a liquid-solid separation compartment or chamber 307 therebetween. The chamber 307 has two portions 307a and 307b defined between vertically spaced conical sections 301 and 302, respectively.

Each of the portions 307a and 307b is provided with an underflow outlet 309 and 311, respectively, positioned at

the apex of the respective conical section, with a combined overflow being withdrawn through an apertured overflow outlet pipe 312 which extends across the vessel and is positioned immediately below the saddle-like intersection of the next higher intersecting conical sections. Each of the portions 307a and 307b is provided with a feed inlet in the form of apertured inlet pipes 314 and 315, respectively, which extend across the vessel at an intermediate portion of each of the portions 307a and 307b.

Each of the inlet pipes, and outlet pipes may be connected to the vessel wall through a suitable bayonet type connection, as shown in Figure 2A.

In operation, the gravity settler functions as described with respect to the embodiment of Figure 1, with liquid-solid feed being introduced into portion 307a through feed pipe 314 and into portion 307b through feed pipe 315. The solids which are intercepted by plates 301 and 302 are withdrawn through outlets 309 and 311, respectively. Solid free overflow is withdrawn through outlet pipe 312.

The use of conical section plates 301 and 302 offers the advantage that all surfaces are sloped towards the underflow withdrawal at the cone apex, which minimizes solid hangup. In addition, the curved shape provides additional mechanical strength, which should eliminate the need for stiffening members.

Although the present invention is particularly suitable for effecting liquid-solid separation of solid particulate material from a coal liquefaction product, it is to be understood that the present invention is suitable for effecting any one of a wide variety of liquid-solid mixtures by gravity settling. Thus, for example, the gravity settler

of the present invention may also be employed for the removal of char fines from oils produced from pyrolysis of coals; finely dispersed solid particles in kerogen retorted from shale, tar sands, oil shale, and the like. The use of gravity settling in accordance with the invention for these applications and others should be apparent to those skilled in the art from the teachings herein.

Although the present invention has been described with respect to the preferred gravity settling apparatus of the present invention, it is to be understood that gravity settling can be effected in accordance with the invention in other forms of apparatus provided with intercepting surfaces for intercepting the solids settling path to cause coalescing of the solids and increase the settling rate.

THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process for separating from a coal liquefaction product solids from a liquid by gravity settling, comprising: introducing a coal liquefaction product into a gravity settler at multiple locations; intercepting the settling path of the solids below each of the feed locations by bringing the solids into contact with inclined intercepting surfaces which divide the gravity settler into separate separation zones to thereby coalesce the solids and increase the solids settling rate; said inclined, intercepting surfaces being at least two vertically spaced, inverted V-shaped plates, each formed of first and second downwardly inclined, upwardly curved, intersecting conical sections having their apices at the vessel wall and defining a substantially enclosed separation zone therebetween; recovering a solids containing underflow from the separate separation zones; and recovering an essentially solids free overflow from the separation zones.

2. The process of Claim 1, wherein the coal liquefaction product includes a promoter liquid which promotes and enhances the separation of solids.

3. A gravity settler for effecting solid-liquid separation, comprising:

a vessel, at least two vertically spaced essentially planar inverted V-shaped plates within the vessel, each of said V-shaped plates being comprised of first and second downwardly oppositely inclined essentially planar sections which extend from the apex of the inverted V-shaped plate to adjacent the vessel wall, said at least two vertically spaced inverted V-shaped plates defining a substantially

enclosed liquid-solid separating chamber therebetween;

inlet means for introducing a liquid-solid feed into said chamber;

first and second underflow outlet means positioned above and adjacent to the lower portions of said first and second sections of the lower of the at least two inverted V-shaped plates; and

overflow outlet means below and adjacent to the apex of the higher of the at least two inverted V-shaped plates for withdrawing clarified liquid from said chamber.

4. The apparatus of Claim 3 wherein the inlet means is comprised of first and second inlet pipes for introducing feed into first and second portions of the chamber above the first and second sections of the inverted V-shaped plates.

5. The apparatus of Claim 3 wherein said first and second sections are inclined at an angle of from 45 to 75° with respect to horizontal.

6. The apparatus of Claim 3 wherein the inverted V-shaped plates are corrugated to provide additional surface for coalescing of solids.

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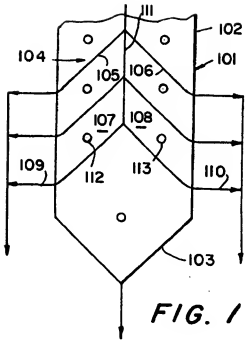

FIG. 1

Rogers Bushin. Pan

FIG. 2

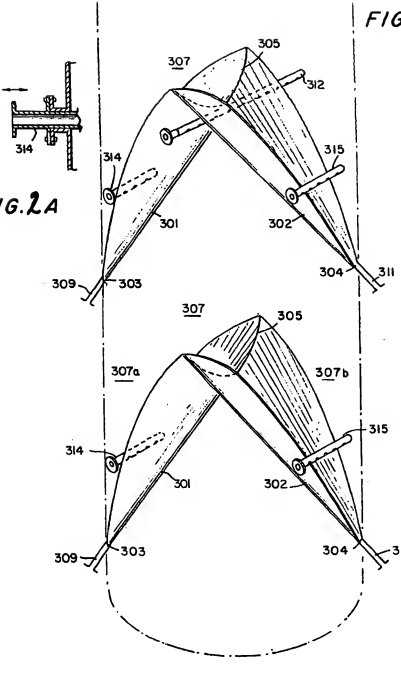

FIG. 2A

Rogers, Bereskin & Parr